LOGIC
Journal of Engineering Design and Technology
Vol. 24 No.1 March 2024 ; p. 31 - 38

p-ISSN : 1412-114X
e-ISSN : 2580-5649
http://ojs2.pnb.ac.id/index.php/LOGIC

ENHANCING ESSENTIAL OIL YIELD: UTILIZING INTERNAL CHOPPER IN STEAM DISTILLATION

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Abstract. Distillation will be optimal if the raw material has a large surface area, for this it requires a chopping process. The aim of this research is to investigate the increase in oil yield achieved through steam distillation with the integration of a chopping module which is a novel distillation process. To achieve this, we employed a distillation reactor equipped with an internal chopping module to enhance both the quantity and quality of Champaca essential oil production. Our experiments involved three variations of pre-distillation treatment: no chopping, chopping inside the reactor, and chopping outside the reactor. The distilled oil was subsequently analyzed using GC-MS. These three chopping methods resulted in varying amounts of Linalool compounds. Unchopped essential materials yielded 6.54% Linalool compounds, while internal chopping produced 32.33%, and external chopping resulted in 7.40% Linalool compounds. The internal chopping method proved to be the most effective in increasing Linalool compound content. Consequently, we can conclude that chopping materials within the distillation reactor during the steam distillation process can increase the main compound's content.

Keywords : Essential Oil, Steam Distillation, Champaca Oil, Chopper

1. INTRODUCTION

As an agricultural country, Indonesia possesses abundant natural plant resources. Among these resources are plants that produce essential oils. There are nearly 150 plants known for their essential oil production, with 40 of them being actively cultivated and exported [1]. Essential oils, also referred to as volatile oils, are extracted from various parts of plants, including leaves, flowers, wood, seeds, or flower buds, through a distillation process. Essential oil extraction methods include steam distillation, distillation, or extraction, which separate the aromatic compounds from the raw materials. The resulting product is an oil containing highly concentrated active compounds, such as aromatic terpenoids and phenolic compounds. This extraction process is considered environmentally friendly as it utilizes water vapor as the solvent [2].

Steam distillation stages include; Preparation of raw materials that have been dried and chopped before being placed into the distillator. A steam vessel is connected separately, delivering hot steam that envelops the surface of the raw material. This steam causes the pores of the raw materials to open, stimulating the release of the oil fraction trapped within. The oil fraction, now released, diffuses with the water vapor and is carried to the cooled condenser, where it condenses into the liquid phase. The oil yield in steam distillation has been reported to be relatively low by previous researchers [3] [4] . To address this issue, some researchers have enhanced their methods, transitioning from super-hot steam distillation [5] to microwave-assisted distillation [6] which are claimed to be more effective in increasing oil yield.

In the essential oil refining industry, steam distillation is a well-established and commonly used method [7]. Increasing production without completely overhauling the method, which would involve acquiring new equipment, consuming additional energy, and preparing the workforce for technology transfer, is a key goal. The

challenge of achieving higher oil yields can often be traced back to the initial treatment of the raw materials. Prior to entering the distillation reactor, raw materials are typically dried to reduce moisture content and then chopped [8]. The chopping process essentially disrupts the tissue structure containing the oil sac, facilitating the oil fraction's diffusion with steam. However, because chopping occurs outside the reactor, some of the oil fraction may be lost prematurely. To prevent the loss of essential oils, the idea is to implement the chopping of raw materials directly within the distillation vessel. This approach ensures that the chopping process occurs simultaneously with the steam flow, resulting in more optimal capture of the oil fraction.

Our study delves into the intricate distillation procedures involved in extracting cempaka oil, a resource plentifully found in Sibang Village, within the Badung Regency of Bali [9]. Despite its abundance, this valuable commodity has remained largely untapped, with minimal processing into essential oil. Through our research, we aim to serve as a pivotal force propelling the development of the local refining industry. By distillation the potential of indigenous resources and unlocking their essence through advanced processing techniques, we pave the way for the conversion of these raw materials into lucrative, high-value products.

2. METHODS

2.1 Material

The test samples used in this study consisted of Champaca *(Magnolia champaca (L.) Figlar)* flowers harvested from Sibang Village, Badung Regency, Bali. Each experiment required 200 grams of orange Champaca flowers. To ensure consistency, all the flowers used were sourced from the same tree. To reduce humidity, all flowers were allowed to wilt in the sun. Subsequently, an electric oven was employed to evenly decrease moisture content throughout the Champaca flowers. The drying process was carried out at 60°C for 2 hours, a temperature chosen based on TGA results indicating that moisture reduction occurred effectively at temperatures below 61.68°C.

2.2 Experimental setup

The experiments using small-scale distillation equipment, comprising three main components: a distillator with a chopper module, a steam reactor, and a condenser. The distillation chamber measures 20x20x40 cm, the schematic is shown in Fig. 1.

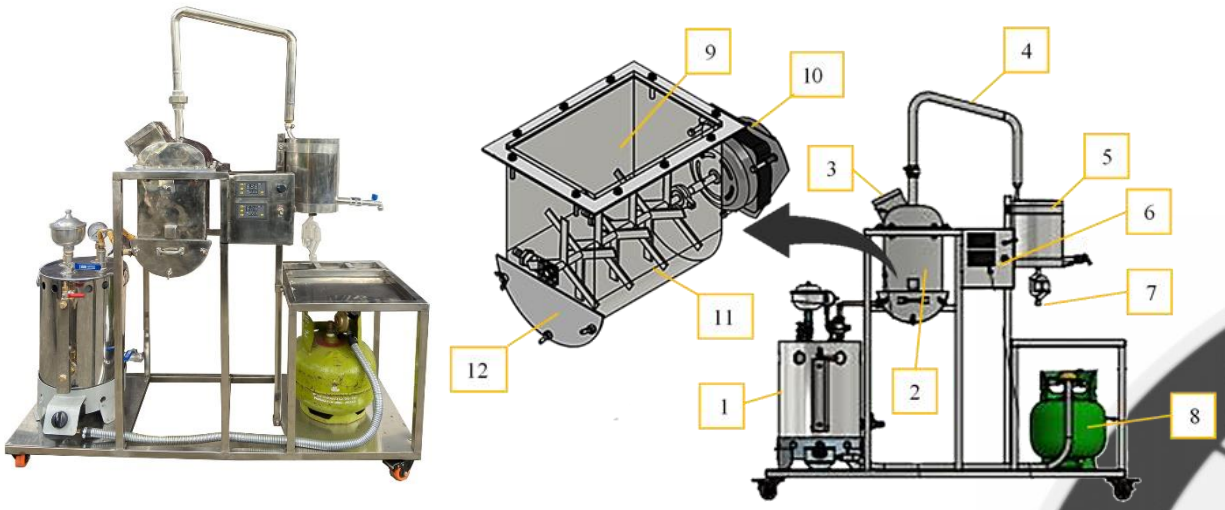


Figure 1. Experimental Setup; 1. Steam Reactor, 2. Distillation, 3. Material Input, 4. Steam Line, 5. Condenser, 6. Control, 7. Oil Output, 8. LPG, 9. Distillation Chamber, 10. Drive Motor, 11. Cutting Blade , 12. Residual output

2.3 Experimental Stage

This study examined the essential oil produced through steam distillation of Champaca flowers, considering three different treatment variations, as illustrated in the diagram, as shown in Fig. 2. The first experiment involved distillation of whole, unchopped Champaca flowers (X1), while the second experiment used Champaca flowers chopped within the distillation chamber (X2). The third experiment used Champaca flowers that were both chopped and dried before distillation (X3). All experiments maintained consistent control variables, including sample type, sample weight, distillation time, and sample size. The quantity of distilled Champaca oil was evaluated based on the percentage of the main component content determined by GC-MS analysis.

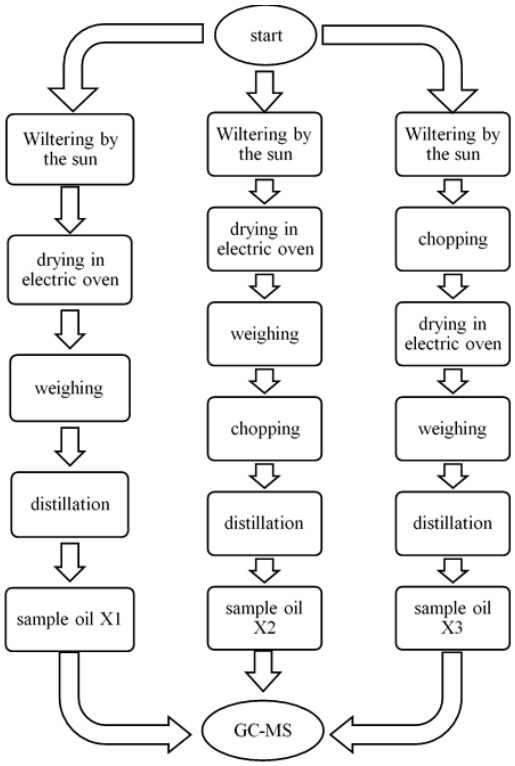


Figure 2. Experiment Stage

2.4 Thermogravimetric Analysis (TGA)

To investigate the biomass behavior under the influence of heat, we conducted a thermogravimetric analysis (TG/DTG) using the TGA-701 Leco instrument, with a measurement precision of ±2°C and a microbalance sensitivity of 0.0001 g. The test sample, comprising 1.0 g of Champaca flower. The sample was subjected to dynamic heating, ranging from room temperature to 950°C, with a heating rate of 20°C/min, all while maintaining a continuous flow of nitrogen gas at 10 mL/min.

2.5 Liquid Analysis

We analyzed the samples using a GC-MS system, which consisted of an Agilent 7890B chromatograph coupled with an Agilent 5977B-MSD spectrometer. The chromatography column employed was an HP-5 MS UI, and Helium served as the carrier gas with a sweep speed of 1.3 mL/min. The heating program was configured to initiate at 70°C for 3 minutes, followed by a ramping rate of 10°C/min until reaching 290°C, which was held for 2 minutes. Compound identification was automated through a comparison of mass spectra with the NIST-14 MS library. The MS scan range spanned from m/z 40 to 350, operating at a frequency of 4.5 scans per second. The gain factor was set at 1.0, and the Electron Multiplier (EM) was maintained at 1780 Volts. The MS source and quadrupole temperatures were set to 230°C and 150°C, respectively.

3. RESULTS AND DISCUSSION

3.1 Champaca Flower Characteristic

The characterization of Champaca flowers involved the microscopic observation of tissues using a Scanning Electron Microscope (SEM). The samples were split on the transverse side for testing, and the results

are presented in Fig. 3. The SEM images reveal that Champaca flower tissues contain pockets or cavities believed to house oil fractions. During the distillation process, steam's heat is employed to deform these tissues, widening them to facilitate the faster release and binding of oil fractions to steam. This process can be more effective if the path traveled by the oil is shorter, thus conserving energy. Shortening this path can be achieved by chopping raw materials before distillation. Therefore, we formulated a provisional hypothesis that chopping the material would lead to an increase in the oil yield.

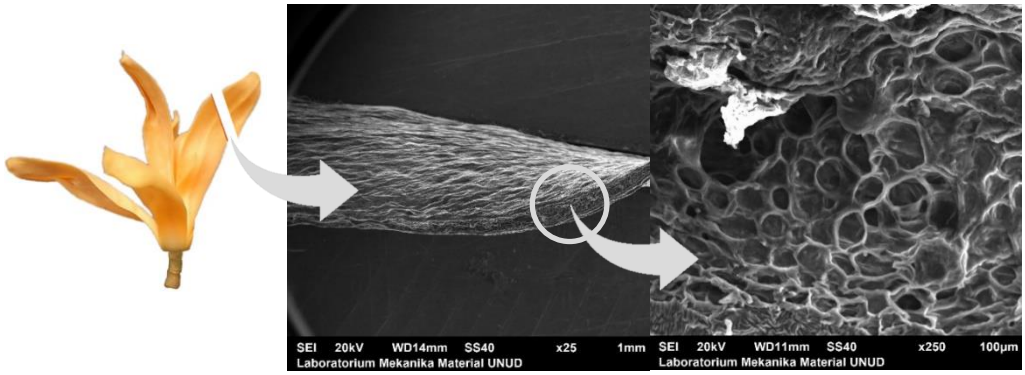


Figure 3. The Transverse Surface Of The Champaca Flower Viewed Using SEM At 25 And 250X Magnification

We conducted proximate analysis through TGA tests to analyze the decomposition process of Champaca flower biomass when subjected to heat. This analysis examines the decomposition of moisture, volatiles, fixed carbon, and ash at specific temperatures corresponding to the applied heat. The test results are presented in Table 1 and visualized in Fig. 4.

Table 1. Proximate Analysis of Champaca Flower

Parameters	%wt*
Moisture	85.41
Volatile (VM)	14.40
Fixed Carbon (FC)	0.13
Ash	0.05

**as received basis (sample was chopped)*

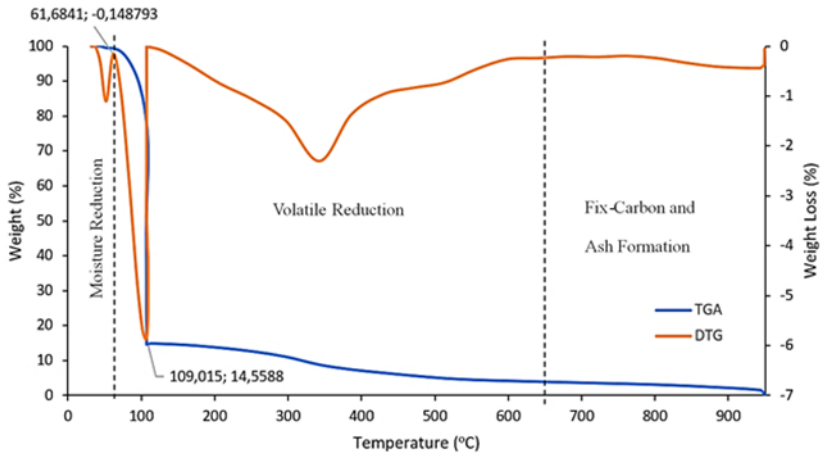


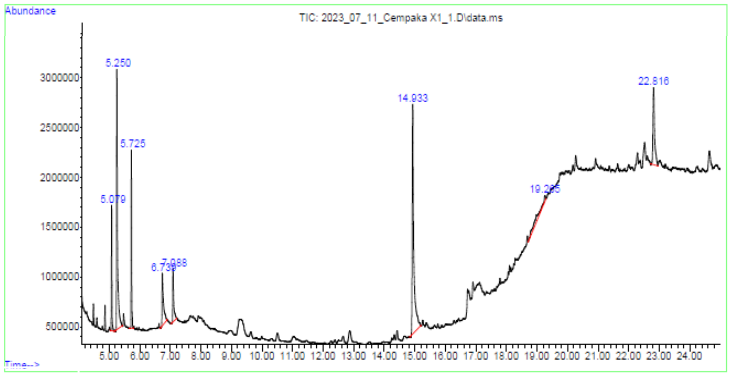
Figure 4. TGA-DTG diagram of Champaca flower

The TGA-DTG graph (Fig. 4) illustrates the reduction in sample mass with increasing heat, corresponding to rising temperatures. Initially, there is a rapid reduction in mass, reaching 85.41% at 109°C. This corresponds to

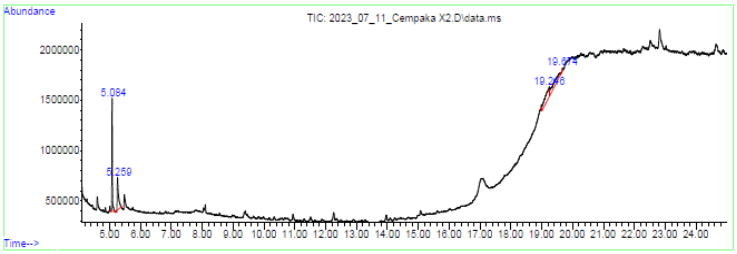
the moisture reduction phase, which typically occurs in the range of 100-110°C, leading to the evaporation of water present in the biomass [10]. Since essential oils have volatile properties, particularly those located outside the flower tissue structure, they are considered part of the water evaporation phase, leading TGA to categorize them as moisture reduction. The DTG graph exhibits a valley that ends at 61.68°C, indicating the initial stage of moisture reduction. To preserve essential oil content, it is advisable to conduct the initial drying stage at temperatures below 61°C, as supported by previous research [11]. The next sharp valley occurs until 109°C, representing a significant reduction in mass attributable to the loss of essential oils. Subsequently, there is a third valley within the temperature range of 109 to 650°C, indicating the evaporation of oil within the tissue structure, accounting for 14.4% of the overall oil content. Finally, there is a minimal mass reduction of 0.13% due to the loss of fixed carbon, leaving behind 0.05% as ash.

3.2 Analysis of Compounds in Distilled Champaca Oil

The distillation experiment yielded three oil samples, denoted as X1, X2, and X3, which were subsequently subjected to GC-MS (Gas Chromatography-Mass Spectrometry) analysis to determine their compound composition. The GC-MS test results are presented in two forms: spectral graphs and a list of compound types (Table 2). In Fig. 5, graph (a) illustrates 8 spectral peaks in sample X1, indicating the presence of 8 dominant types of compounds within this sample. Sample X2, shown in graph (b), exhibits 4 dominant spectra, signifying the presence of 4 dominant compound types. Meanwhile, sample X3, depicted in graph (c), displays 8 dominant spectra. The comparison of peak spectral height and retention time serves as the basis for identifying and categorizing compound types, referencing the NIST Chemistry Web-Book catalog [12].



(a)



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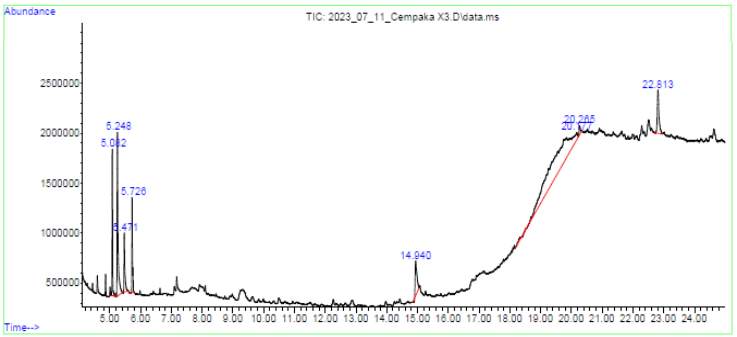

(c)

Figure 5. GC-MS Spectrum Graph: (a) X1, (b) X2 dan (c) X3

Linalool, a key component of essential oils, particularly Champaca oil, is represented by the molecular formula $C_{10}H_{18}O$ [13]–[15] with the molecular formula $C_{10}H_{18}O$. In this study, the quantity of Linalool was employed as an indicator to assess the distillation process's performance. Table 2 presents the column area section, showing the percentage of Linalool content in each sample. Sample X1 contains 6.54% Linalool, while sample X2 boasts the highest concentration at 32.33%, and sample X3 contains 7.4% Linalool. Notably, sample X2 exhibits the highest Linalool content, suggesting that the distillation process with internal chopping yields Champaca oil of superior quality compared to the other two methods.

Table 2. List Of Compounds in Champaca Oil Based On GC-MS Test Results

PK#	RT	Area%	Library/ID	Ref#	CAS#	Qual
			Sample : X1			
1	5.079	6.54	Linalool	27451	000078-70-6	91
2	5.250	22.85	Phenylethyl Alcohol	10108	000060-12-8	94
3	5.725	9.11	(3R,6S)-2,2,6-Trimethyl-6-vinyltetrahydro-2H-pyran-3ol	39780	039028-58-5	72
4	6.739	6.96	Indole	8731	000120-72-9	95
5	7.088	4.90	Methyl anthranilate	25546	000134-20-3	95
6	14.933	33.24	n-Hexadecanoic acid	117418	000057-10-3	99
7	19.265	4.49	Tetrasiloxane, decamethyl-	168283	000141-62-8	50
8	22.816	11.90	Acetic acid, [4-(1,1-dimethylethyl)phenoxy]-,methyl ester	85348	088530-52-3	47
			Sample : X2			
1	5.084	32.33	Linalool	27447	000078-70-6	96
2	5.259	20.66	Phenylethyl Alcohol	10107	000060-12-8	94
3	19.246	25.90	Tris(tert-butyldimethylsilyloxy)arsane	260810	1000366-57-5	52
4	19.674	21.12	Arsenous Acid, tris(trimethylsilyl) ester	199618	055429-29-3	52
			Sample : X3			
1	5.082	7.40	Linalool	27447	000078-70-6	96
2	5.248	14.31	Phenylethyl Alcohol	10108	000060-12-8	94
3	5.471	4.78	Benzyl nitrile	8741	000140-29-4	96
4	5.726	5.56	(3R,6S)-2,2,6-Trimethyl-6-vinyltetrahydro-2H-pyran-3ol	39780	039028-58-5	83
5	14.940	5.38	n-Hexadecanoic acid	117419	000057-10-3	97
6	20.177	54.72	2" -Hydroxy-5' -methylacetophenone, TMS derivative	85149	97389-69-0	50
7	20.265	1.02	2,4,6-cycloheptatrien-1-one,3,5-bis-trimethylsilyl-	111265	1000161-21-8	50
8	22.813	6.83	1,2-Bis(trimethylsilyl)benzena	85160	017151-09-6	50

The distillation of whole Champaca flowers yields the lowest Linalool content and also includes seven other types of compounds, such as acids (45.14% - including n-Hexadecanoic acid and Acetic acid), alcohols (22.85%), and aromatic hydrocarbon compounds (25.46% - including Trimethyl, Indole, Methyl anthranilate, and Tetrasiloxane). The presence of these additional compounds is attributed to the excess heat absorbed by the flower tissues during distillation. This occurs when the trapped oil has to traverse a lengthy trajectory, causing it to decompose due to the heat into various other compounds. High temperatures promote chemical reactions between compounds, leading to the formation of new compounds. A similar pattern is observed in sample X3, where the distillation process initiates with the drying of chopped samples, causing the rapid evaporation of Linalool, leaving only a minimal amount. Elevated temperatures further promote the decomposition of tissue components into hydrocarbon compounds, constituting 72.91% of the total composition. These hydrocarbon compounds include Benzyl nitrile, Trimethyl, methylacetophenone, cycloheptatrien, and benzena. Additionally, alcohol compounds make up 14.31% of the composition, followed by acids at 5.38%. The distillation process using the internal chopping method effectively retains Linalool components within the tissue pockets. When exposed to steam, these components readily escape and diffuse with the steam, as illustrated in Fig. 6. Because Linalool compounds are positioned internally, they can flow shorter distances. It can be inferred that smaller cutting sizes result in higher

Linalool compound content in the oil. This observation aligns with findings from previous researchers [16], [17], although they employed external chopping methods before the distillation process.

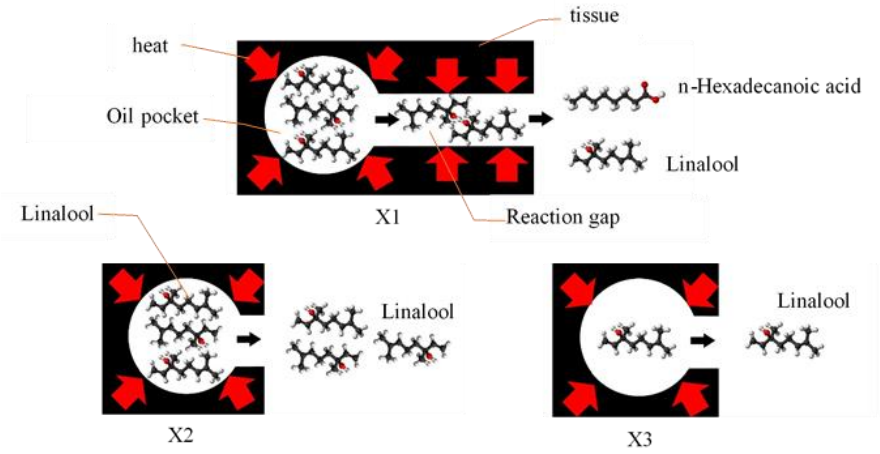

Figure 6. Reaction scheme and release of compounds in tissues

In addition to Linalool, the internal enumeration method yields alcohol compounds (Phenylethyl Alcohol) at a concentration of 20.66% and acids at 47.02% (including arsane and Arsenous Acid). Both of these compound groups are formed due to the presence of excess oxygen, particularly in the steam containing oxygen. Notably, compared to the other two methods, the internal chopping method produces only three additional types of compounds aside from Linalool. This suggests that the subsequent purification process will be more straightforward and efficient.

4. CONCLUSION

Linalool emerges as the predominant compound in champaca oil obtained through steam distillation. Varied chopping methods employed during the distillation process result in differing concentrations of Linalool compounds. Specifically, unchopped essential materials yield Linalool compounds at a rate of 6.54%, internal chopping produces 32.33%, and external chopping yields 7.40% Linalool compounds. The volatile nature of essential oils underscores the necessity for precise distillation techniques. The internal chopping method notably yields the highest concentration of Linalool compounds. Hence, it can be inferred that incorporating internal chopping of materials within the distillation reactor effectively enhances the main compound content during steam distillation processes.

5. ACKNOWLEDGEMENT

This research was funded by Politeknik Negeri Bali, based on contract No. 1733/PL8/AL.04/2023, year 2023.

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